

Standard Operating Procedure for GLNPO Board Analyses

LG500

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Standard Operating Procedure for GLNPO Board Analyses

1.0 SCOPE AND APPLICATION

- 1.1 This method is a description of the procedure used on the EPA Monitoring vessel, the *R/V Lake Guardian*, and includes the determination of Conductivity, Total Alkalinity as CaCO₃, Turbidity and pH.
- 1.2 Dissolved Oxygen, when determined, is analyzed at nearly the same time as the subject analyses, but is described in another SOP.

2.0 SUMMARY OF METHOD

- 2.1 At the beginning of each lake cruise, applicable instruments, used for the water quality analyses of lake water samples, are initially calibrated and control standards analyzed.
- 2.2 For routine sample analyses, a sample is placed on the conductivity apparatus and heated to 25.0 °C. The conductivity reading is recorded, after which the pH electrode is placed in the sample and that reading is recorded. The beaker is removed from the apparatus, and the beaker contents are used to fill the turbidity cuvette and the 100-mL alkalinity sample volume measuring flask. The contents of the alkalinity flask are transferred to the alkalinity apparatus and the titration is performed. The readings from the alkalinity titration and the turbidimeter are recorded.
- 2.3 A set of control standards is run after the last station sampled on each 12-hour shift.
- 2.4 There is one board chemistry analytical batch per each 12-hour shift. Each batch is recorded with a unique batchID and contains results for RFS samples and their associated control standards, field duplicates, and field reagent blanks (pH is not run on field reagent blanks).

3.0 SAMPLE HANDLING AND PRESERVATION

- 3.1 Samples are collected in new one-gallon cubitainers (polyethylene plastic collapsible containers) or new one-half gallon polyethylene milk cartons (See LG 200, *Field Sampling Using the Rosette Sampler*).
- 3.2 Analyses are routinely performed within two hours of collection. The cubitainers are retained refrigerated for the duration of the 12-hour shift, so that, if control standards show a system to be out of control, it can be brought back into control and the samples re-analyzed for that parameter(s) (See Appendix B, *Quality Assurance Project Plan for the Great Lakes Water Quality Surveys*).

4.0 INTERFERENCES

- 4.1 No major interferences are known with regard to these analyses with Great Lakes water samples.

5.0 APPARATUS

- 5.1 Conductivity Meter, with nominal 1-cm cell constant (YSI Model 3200 with YSI probe 3253).
- 5.2 Variable speed stirring motor with glass stirring paddle. Two each.
- 5.3 Immersion heater with proportional temperature control and bypass on/off control.

- 5.4 Thermometer with 0.01 °C subdivisions, YSI Model 4600
- 5.5 Meter pH (Fisher Accumet Model 15) with Ross combination Electrode and automatic temperature compensation two each meter and electrode.
- 5.6 HACH turbidimeter Model 2100N
- 5.7 Burette, 25-mL automatic fill to zero, or Digital valveless burette.
- 5.8 The conductivity apparatus consists of: the Meter with a conductivity electrode; an immersion heater, a small electric motor, with a speed controller, driving a stirring paddle; a thermometer with 0.01 °C divisions; a small table to hold the sample beaker such that the thermometer, the electrode, the stirring paddle, and the immersion heater are immersed in the sample.
- 5.9 The alkalinity apparatus consists of: a pH meter and electrode; a small electric motor with a speed controller, driving a stirring paddle; a burette; and a small table to hold the sample beaker such that the electrode and stirring paddle are immersed in the sample with the burette tip draining into the sample beaker.

6.0 REAGENTS

- 6.1 Reagent water: All reagents are prepared using water that has passed through two ion exchange cartridges, an organic removal cartridge and a 0.2-micron filter. The Conductivity of this reagent water used for standard preparation is less than 0.1 µmhos/cm (10 megohm-cm). The feed water to this purification system is from a polyethylene tank fed from a 'Super Still' (a vacuum distillation system maintained by the engineering staff).
- 6.2 Conductivity
 - 6.2.1 Stock sodium chloride set standard solution. 3.718g/L of Commercial NaCl Standard.
 - 6.2.2 Working Calibration Standards
 - 6.2.2.1 The following are prepared with volumetric lab ware. Two liters of each of these standards are prepared prior to the survey. A calibration standard wash will be maintained for each of the calibration standards the same as is done with the control standards.

mL of prepared NaCl solution diluted to 1 liter	Approximate Conductivity $\mu\text{mhos/cm}$
50	387.14
40	310.93
30	234.25
20	156.99
10	79.02

6.2.3 Stock Conductivity Control Standard Solution: Prepared from 0.1 Molal KCl stock solution.

6.2.4 Control Standards: The following are prepared using volumetric lab ware.

QC Type	mL 0.1 Molal KCl stock diluted to 1 Liter	Conductivity $\mu\text{mhos/cm}$ at 25°C
Low Control Standard (CH)	7	102.9
High Control Standard (CL)	20	290.6

6.2.4.1 One liter of each of these working control standards is prepared as needed.

6.3 Turbidity

6.3.1 Formazin working standards

Prior to each survey, two solutions are prepared. Shelf life is one month.

5.00 gm/400 mL hydrazine sulfate, $(\text{NH}_2)_2\text{H}_2\text{SO}_4$

50.00 gm/400 mL hexamethylenetetramine

6.3.2 The two solutions are brought to 25°C are mixed in a clean dry 1 Liter volumetric flask, and dilute to volume with reagent water. This solution is allowed to stand 24 hours at 25°C. Shelf life is one month. Nominal turbidity is 4000 NTU.

6.3.3 The 4000 NTU stock solution is diluted to create a 100 NTU solution by adding 100mL of reagent water to a clean 200 mL volumetric flask then add 5 mL of well mixed stock solution and dilute to 200mL with reagent water. Two dilutions are prepared from the 100 NTU solution and used as control standards for the turbidimeter. The control standards are run after the last station on each shift.

aliquot of 100 NTU diluted to 1 Liter with reagent water	Final NTU value
100 mL	10
5 mL	0.5

6.3.4 The calibration standards for turbidity are (StablCal™) stabilized formazin turbidity standards obtained from HACH. They are >0.1, 1, 10, 100, 1000 NTU. The instrument is calibrated at the beginning of each survey. Calibration should be stable for the entire survey so the initial calibration should be repeated however many times it takes to get it right. Solid turbidity standards are maintained for the HACH meter and readings are obtained at the beginning of each survey.

6.4 pH

6.4.1 Calibration standards for pH. Commercial pH standards (10, 7 and 4) are used for the pH standardization.

6.4.2 Commercial buffer packets (6.86 and 9.18) are used to prepare the pH check standards.

6.5 Preparation of 0.02 N sulfuric acid solution.

6.5.1 Commercial standard 1.00 N sulfuric acid is diluted 20 mL per liter with reagent water.

6.6 Alkalinity

6.6.1 $\text{Na}_2\text{CO}_3 \cdot 0.1 \text{ N}$. The contents of a Dilutit Commercial Standard capsule are diluted to one liter or a 0.1 N solution is prepared from a 1 N commercial standard.

6.6.2 Total Alkalinity Control Standards

Concentration	mL/L of 0.1 N Na_2CO_3
100 mg/L T.A. as CaCO_3	20
40 mg/L T.A. as CaCO_3	8

7.0 INSTRUMENT CALIBRATION

7.1 Conductivity

- 7.1.1 The same YSI Model 35 and Model 32 conductivity meters and electrode were used by this program for more than 5 years with only occasional calibration. The high and low control standards were within an acceptable range throughout that period. Beginning with the spring cruise of 2004, YSI Model 3200 with YSI Model 3253 Conductivity Cell was brought into service.
- 7.1.2 Prior to the first lake of each survey, the calibration is performed by tracking through the menu to Multi point standardization and heating each standard to 25 degrees. (OPERATION→CELL→CAL K→MULTI POINT→) On the next lake of the cruise, the calibration is checked for one of the standards. If the standards produce results at that time that differ by more than 1 % from the standardization, the instrument is re-standardized prior to the next lake, until each standard from two consecutive series differ by no more than 1 %.
- 7.1.3 Calibration of the Model 3253: This calibration is complex. It may be necessary to perform this calibration multiple times before becoming comfortable with the result. The meter must be calibrated with material at exactly 25 degrees C. The samples can be run anywhere around 25 degrees provided that the temperature has stabilized such that the temperature probe and the conductivity probe are both at the same exact temperature. The procedure of sequencing through a wash and a standard (section 10.3) is used for calibration as well as control standards. The configure menu is used to set the parameters for running samples. Calibration can be performed independently of the configure menu except that the configuration number must be selected. Multipoint calibration and single point calibration can be performed on each of the 6 configurations. The configuration menu can then select any one of the twelve for sample analysis. When determining conductivity of samples by multipoint calibration the K displayed is replaced by Km and this Km is interpolated from the multiple standards. The current operating mode of the instrument is indicated in the upper right hand corner of the display. By pressing the mode key repeatedly, the mode will change from Data to Operation and back to Data. When in the operation mode, a soft key will allow one to navigate to the calibrate or configure menu. To get back to Data mode simply press the mode key until Data is displayed in the upper right corner.

To get to calibrate mode:

- 1) Press mode to get to operation(top right corner)
- 2) Press cell soft key
- 3) Press config soft key
- 4) Select configuration number (0-5) using the up and down soft key
- 5) Press mode to get back to cell
- 6) Then press cal soft key
- 7) Then press mult soft key
- 8) The current standard is indicated on line 3 (0 to 4).
- 9) The value of each digit of the current standard is adjusted by the up or down key.
The digit that is currently adjustable has a blinking underline
- 10) Digit soft key is used to move the blinker to the next digit.
- 11) When the value of the current standard is entered and the standard is on the apparatus stable at 25 degrees centigrade the K value on line 2 should be very close to 1.00.
- 12) Press the enter soft key to load the factor. Line 3 should go from 0 to 1 indicating that it is ready to accept the next standard.
- 13) Repeat this procedure for the other four standards
- 14) After which press the mode until you get to cell.
- 15) Press configure soft key and select the configuration number that you calibrated. The configuration number displayed is the one that was calibrated.
- 16) Select mult calib and natural temperature compensation.

- 17) Press mode until you get to Data mode.
- 18) The K displayed should be Km.

The proof of the procedure is to analyze the five standards in the Data mode. The cell calibration is very stable. It should not be necessary to perform the calibration on each lake. One calibration standard should be run in the data mode along with the control standards at the beginning of each lake. For additional information refer to the (YSI Model 3200 Operations Manual) located in the wet lab on top of the medical locker. Section 3 Pages 12 – 15 have detailed calibration instructions

7.2 pH

7.2.1 This paragraph describes the use of the AR15 pH meter for day-to-day use. To initially set up the meter go to the operation manual for these instruments. The meter for pH determination is standardized at the beginning of each shift with buffers 7 and 10. In the following description, stable readings should be obtained before proceeding to the next step. Each step is numbered. Standardize at the beginning of each shift.

- 1) Sequence the electrode through lake water, buffer 7 wash, and buffer 7 standard.
- 2) Record the pH and temperature of the buffer 7 standard. At this point, the meter should be displaying the pH icon, icons for buffers 7 and 10, and the pH should be displayed to two decimal places.
- 3) Press “setup” twice. “Clear buffer” should be displayed to the left of the beaker icon, “7 10” to the right.
- 4) Press Enter. The area where the buffer icons are displayed should be blank. The uncalibrated value of the pH 7 buffer is displayed.
- 5) Press STD. The buffer group 2 4 7 10 12 should be displayed briefly.
- 6) Press STD again. The buffer icon “7” should appear in the buffer icon area. The pH reading should change to the pH 7 buffer value at the temperature of the standardization.
- 7) Sequence the electrode through lake water, buffer 10 wash, and buffer 10 standard.
- 8) Press STD. The pH of the pH 10 buffer is displayed assuming an efficiency of 100%.
- 9) Press STD again. The actual electrode efficiency should be displayed briefly and a 10 should join the 7 to the right of the beaker icon.

Unlike the previous meter, the electrode efficiency is not retained between standardizations. Thus, after a one-point standardization, the meter uses 100 as the efficiency until a second standard is entered, regardless of the actual value.

If the buffers are not cleared prior to standardization, “ELECTRODE ERROR” may appear as result of a slight drift in the electrode potential. This is not an indication that the electrode is bad. It just means that the instrument must be standardized using the proper SOP.

7.2.2 To initially set up the AB15 or AR15, consult the instrument operations manual.

7.3 Alkalinity

- 7.3.1 The Model 15 AB pH Meter used for the Alkalinity titration is calibrated using the onscreen instructions and pH buffers 4 and 7. The previous calibration is cleared prior to calibrating at the beginning of each shift.

7.4 Turbidity

7.4.1 HACH CALIBRATION

- 1) Use UNITS/EXIT button to light up NTU
- 2) Use signal averaging
- 3) Use Ratio
- 4) Use AUTO RANGE
- 5) Press CAL ---- Displays last blank water STD reading and S0 lights up
- 6) Load cell with reagent water
- 7) Press Enter
- 8) Wait for count down from 60 to 0
- 9) Displays next expected STD '10' S1 lights up
- 10) Load STD 10
- 11) Press Enter
- 12) Wait for count down from 60 to 0
- 13) Displays next expected STD '100' S2 lights up
- 14) Load STD 100
- 15) Press Enter
- 16) Wait for count down from 60 to 0
- 17) Displays next expected STD '1000' S3 lights up
- 18) Load STD 1000
- 19) Press Enter
- 20) Wait for count down from 60 to 0
- 21) Displays next expected STD '1.000' S4 lights up
- 22) Load STD 1.0
- 23) Press Enter
- 24) Wait for count down from 60 to 0
- 25) Displays last reagent water STD reading and S0 lights up
- 26) Press CAL Meter reverts to normal use with new standardization.

When attempting to do STDs in order '0.1, 1, 10, 100, 1000', error message 02 is displayed and standardization is aborted. For additional information refer to (HACH Model 2100N Turbidimeter Instruction Manual) located in the wet lab on top of the medical locker Section 3 pages 33-45 have detailed calibration instructions.

8.0 ANALYTICAL PROCEDURE

8.1 General considerations

- 8.1.1 To preserve the integrity of the sample, the Cubitainer/(bulk sample container) must be agitated before withdrawing an aliquot.
- 8.1.2 Rinsing of the conductivity equipment or the alkalinity volumetric flask is not necessary between samples from the same lake water station. It is necessary however to rinse the equipment prior to readings from the standards and field blanks.
- 8.1.3 The pH measuring electrode should be maintained in lake water except during measurements.

8.1.4 It is not necessary to rinse the 4.5 pH solution off the alkalinity equipment between titrations.

8.2 At each station:

- 1) A fresh lake water sample is placed on the conductivity apparatus and the temperature is raised to 25°C. This water is then used to rinse the alkalinity volumetric flask and to fill the receptacle for the pH electrode.
- 2) A fresh aliquot is then poured into the conductivity beaker so that the beaker is full to within ¼ inch of the top. The beaker is then placed on the conductivity apparatus and temperature raised to 25.0°C ± 1°C while the stirrer is operating to produce rapid circulation without breaking the surface of the sample.
- 3) When the temperature and conductivity readings are stable (this may take some time) at 25.0°C ± 1°C, the conductivity is recorded, and the pH electrode is inserted into the sample. Do not attempt to determine the pH of reagent water (i.e., the field blank).
- 4) When the pH meter indicates that the pH reading is stable, the pH reading is recorded, and the pH electrode is returned to the pH electrode receptacle.
- 5) The beaker is removed from the conductivity apparatus and portions are used to fill the alkalinity sample volume measuring flask and the turbidity cuvette.
- 6) The 100 mL from the alkalinity volumetric flask is placed on the alkalinity apparatus and the stirring speed is adjusted to achieve rapid circulation without breaking the surface of the sample. Titration to pH 4.5 (plus or minus one drop) is performed. The total alkalinity (burette reading x ten (10)) is recorded.
- 7) The turbidity is recorded as soon as the reading becomes stable.
- 8) Subsequent lake water samples from the same station are treated as from step 2 above.
- 9) Prior to analyzing the field blank the conductivity apparatus, the 100 mL flask and the turbidity cuvette are rinsed with field blank before making the measurements. No pH measurement is made on the field blank. After analysis of any sample other than lake water, the equipment (except the alkalinity beaker and electrode) must be rinsed with lake water before proceeding.

8.3 When the apparatus is not being used, the conductivity cell is immersed in a lake water sample. The pH electrode is immersed in a lake water sample. The alkalinity pH electrode is immersed in the last titrated sample. The turbidity cuvette is filled with reagent water. For extended periods of non-use, see section 11.1.

9.0 CALCULATIONS

9.1 The conductivity is read directly from the meter.

9.2 The pH is read directly from the meter.

9.3 The alkalinity is 10x the burette reading.

9.4 The turbidity is recorded directly from the meter.

10.0 QUALITY CONTROL

10.1 The Winkler determination is used for quality assurance purposes for the SeaBird determination of dissolved oxygen. It will be run in duplicate on one depth from approximately three predesignated stations per lake on the non-DO cruises. On the Lake Erie DO cruises, it will be performed in duplicate on the surface and the B- samples

at each station. An oxygen saturated water sample will be analyzed by Winkler at least once per lake on the non-DO cruises and once per shift of the Lake Erie DO cruises. Refer to Appendix D, *Sampling and Analytical Procedures for GLNPO's Open Lake Water Quality Survey of the Great Lakes*, for DO and temperature profiles for this survey.

- 10.2 During sampling, High and Low Control Standards are analyzed after the last station on each 12- hour shift for each of the four parameters. If any standard exceeds the warning range, the sample values and control values are recorded, but the system is not used again until the parameter is re-standardized and the control standards have verified that the system is in control. If any standard exceeds the control limit, the parameter is re-standardized and all samples analyzed since the last valid control standards are re-analyzed along with the control standards. Appropriate information is inserted in the remarks column of samples associated with control standards beyond the control limits or beyond the holding time. If after re-standardization, the control standards are not within normal range, corrective action must be taken before proceeding with further analyses. All analyses (of samples and controls) are recorded regardless of whether the control standards are within limits or not.

10.3 Conductivity

- 10.3.1 Each conductivity standard consists of unused standard and a bottle holding the 'wash.' Each time a standard is to be used, the 'wash' is placed on the apparatus and the stirrer is activated to allow the apparatus to equilibrate with that wash. That wash is then discarded and a fresh aliquot of standard is placed on the machine. After the calibration or measurement, this material is then placed in the 'wash' container for use as a wash the next time the standard is used. Do not put anything in the standard bottle, do not return 'wash' to the wash bottle, do not discard standard. Do place used standard in the empty wash bottle.

- 10.3.2 The following QA samples must be prepared and analyzed at the minimum frequency indicated.

QC Type	Frequency	Acceptance Criteria (µmhos/cm)
High Control Standard (CH)	At the onset, starting with the initial calibration of instruments for each lake survey, and after the last station on each 12-hour shift	288.6 - 292.6 (warning) 287.6 - 293.6 (control)
Low Control Standard (CL)	At the onset, starting with the initial calibration of instruments for each lake survey, and after the last station on each 12-hour shift	72.2 - 75.2 (warning) 71.7 - 75.7(control)
Field Reagent Blank (FRB)	One per basin ^a	2µmho
Field Duplicate (FD1)	One per basin ^a	Difference 2.0

^a A field duplicate, and field reagent blank are collected with each group of 3, 4, or 5 stations depending on the lake. A Random Number Generator (RNG) is used to determine the stations and depths of these QA samples. Where basins are well defined, at least one of each is collected from each basin.

10.4 Alkalinity

10.4.1 The sample volume measurement device for the alkalinity determination must be rinsed with 10 to 20 mL of the appropriate standard or blank prior to use for measuring volume of standard or blank. The titration vessel should never be rinsed with standard or sample. After the pH electrode for the alkalinity is standardized, the electrode should be thoroughly rinsed with reagent water. Thereafter, the titration vessel need not be emptied after each titration until the next use. Neither the electrode nor the vessel needs to be rinsed before the next use, since it contains pH 4.5 material. If the endpoint is overshoot, then both the electrode and vessel should be rinsed with reagent water prior to the next attempt. The sample volume measurement device need not be rinsed between samples from the same lake water station, but it should be rinsed with about 20 mL of sample before use for the first sample. The sample volume measurement device should be rinsed with the appropriate material prior to being used to measure a volume of FRB or Control

10.4.2 The following QA samples must be prepared and analyzed at the minimum frequency indicated.

QC Type	Frequency	Acceptance Criteria (mg/L)
High Control Standard (CH)	At the onset, starting with the initial calibration of instruments for each lake survey, and after the last station on each 12 hour shift	98 - 102 (warning) 97 - 103 (control)
Low Control Standard (CL)	At the onset, starting with the initial calibration of instruments for each lake survey, and after the last station on each 12 hour shift	38.5 - 41.5 (warning) 38 - 42 (control)
Field Reagent Blank (FRB)	One per basin ^a	3
Field Duplicate (FD1)	One per basin ^a	2

^a A field duplicate and field reagent blank are collected with each group of 3, 4, or 5 stations depending on the lake. A Random Number Generator (RNG) is used to determine the stations and depths of these QC samples. Where basins are well defined, at least one of each is collected from each basin.

10.5 pH

10.5.1 The electrode for pH determination is maintained in a vessel of lake water. The calibration and control standards for pH are retained in 125-mL bottles labeled with the pH value and either standard or wash. At the beginning of each lake survey, i.e., 'Michigan spring 2000,' the 'wash' is discarded and the 'standards' become the new 'wash.' Fresh material is then used for the 'standards.' Each time a standard is used, the electrode is immersed in the 'wash' prior to immersion in the 'standard.' After a 'standard' is used, the electrode should be rinsed by immersion in lake water or reagent water. If the vessel that is used for electrode retention is used for rinsing between standards, it should be filled with fresh lake water before the next lake sample determination. Do not attempt to measure the pH of reagent water except in the alkalinity determination. While the true total alkalinity end point for reagent water is not 4.5, we will use that as the end point for simplicity and to be consistent.

10.5.2 The following QC samples must be prepared and analyzed at the minimum frequency indicated.

QC Type	Frequency	Acceptance Criteria (SU)
High Control Standard (CH)	At the onset, starting with the initial calibration of instruments for each lake survey, and after the last station on each 12-hour shift	9.18 ± 0.2 (warning) 9.18 ± 0.3 (control)
Low Control Standard (CL)	At the onset, starting with the initial calibration of instruments for each lake survey, and after the last station on each 12-hour shift	6.86 ± 0.2 (warning) 6.86 ± 0.3 (control)
Field Duplicate (FD1)	One per basin ^a	Difference 0.3

^a A field duplicate and field reagent blank are collected with each group of 3, 4, or 5 stations depending on the lake. A Random Number Generator (RNG) is used to determine the stations and depths of these QC samples. Where basins are well defined, at least one of each is collected from each basin.

10.6 Turbidity

10.6.1 When taking measurements, the outside of the turbidity cuvette must be clean and dry. It is never touched with bare hands. To preclude spilling water on the outside of the cuvette, the alkalinity sample volume measuring flask can be used to fill the cuvette. The turbidity cuvette cap is marked with an arrow on the lid to allow the cuvette to be placed in the instrument in a repeatable position. The arrow is directed toward the back of the instrument when taking readings. All readings, with the exception of the STABLCAL calibration standards should be taken using the same cuvette. It is not necessary to rinse the turbidity cuvette between lake water samples, except where there is a pronounced difference between two samples.

10.6.2 The following QA samples must be prepared and analyzed at the minimum frequency indicated.

QC Type	Frequency	Acceptance Criteria (NTU)
High Control Standard (CH)	At the onset, starting with the initial calibration of instruments for each lake survey, and after the last station on each 12-hour shift	10 ± 1 (warning) 10 ± 2 (control)
Low Control Standard (CL)	At the onset, starting with the initial calibration of instruments for each lake survey, and after the last station on each 12-hour shift	0.5 ± 0.2 (warning) 0.5 ± 0.3 (control)
Field Reagent Blank (FRB)	One per basin ^a	0.15
Field Duplicate (FD1)	One per basin ^a	Difference ($.2 + 0.1 \times$ mean reading)

^a A field duplicate and field reagent blank are collected with each group of 3, 4, or 5 stations depending on the lake. A Random Number Generator (RNG) is used to determine the stations and depths of these QC samples. Where basins are well defined, at least one of each is collected from each basin.

10.7 Corrective Action

- 10.7.1 Corrective action procedures will often be handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, calibration mixes, instrument sensitivity, and any other potential sources of error. If failure occurs and an error is identified, the analyst should re-run quality control and RFS samples in the entire analytical batch to confirm the results. Because analysis of field duplicates usually occurs after leaving a specific station and re-sampling is largely impossible, re-analysis of these samples to confirm results may be the limit to corrective actions when all other QC samples within a batch meet acceptance criteria. For analyses conducted onboard, if the problem persists or cannot be identified, the matter must be referred to the Chief Scientist for further investigation. Depending upon the Chief Scientist's evaluation, the analyst may or may not be required to re-run the samples. Once a decision is made, full documentation of the corrective action procedures and assessment of the final result must be filed with the WQS QM Technical Lead or the GLNPO QM.

10.8 Data Reporting/Recording

- 10.8.1 When corrective actions are not feasible or do not resolve QC failure, the analyst is responsible for identifying all failed QC samples and RFS samples. The analyst should document the QC information on the hard-copy Field Information Recording Forms (Appendix H).
- 10.8.2 There is one board chemistry analytical batch per each 12-hour shift. If, for some reason, there are multiple analysts and/or a new set of control standards are utilized during a 12-hour shift, a new batch should be initiated. Prior to the start of each survey, a group of board chemistry batchIDs are loaded into the GLEND data entry tool. Following each shift, it is the responsibility of the analyst to use the next consecutive batchID. When recording results, the board chemistry batchID scheme is as follows:

YYPPnnn	
YY	Year: Two-digit years such as "00," "01," "02"
PP	Parameter group: "BD" representing board chemistry
nnn	Numerical sequence such as "001," "002," "003"

11.0 PREVENTIVE MAINTENANCE

- 11.1 During surveys, the pH and conductivity electrodes should be kept immersed. Between surveys, the conductivity electrode should be thoroughly rinsed in reagent water prior to allowing it to dry. The pH electrodes should be immersed in pH 7 buffer between surveys so that the glass membrane remains immersed. The level of KCl solution in the reference electrode should be checked at the beginning of each Lake survey and filled to within ¼ inch of the filling hole.
- 11.2 The turbidity cell should be thoroughly rinsed with reagent water at the conclusion of each survey.

12.0 TROUBLESHOOTING/CORRECTIVE ACTION

- 12.1 If the conductivity cell is so dirty that bubbles always form at the top of the cell when a receptacle of water is placed on the apparatus, ethanol or 1 N NaOH may be used to attempt cleaning. Neither should be left on the cell for more than two or three minutes.

13.0 SAFETY AND WASTE HANDLING

- 13.1 Refer to GLNPO's *Health, Safety and Environmental Compliance Manual* (May 1997, or as amended) and individual instrument procedural operations manuals for specific details on applicable 1) personal health and safety issues; 2) instrumental, chemical, and waste handling procedures; and 3) accident prevention. This applies to all EPA personnel, EPA contractors or federal, state, or local government agencies, and persons who operate or are passengers onboard US EPA GLNPO vessels during all activities and surveys.
- 13.2 All containers storing reagents, standards, controls, blanks, and wastes used in the laboratory must be properly identified through appropriate labeling and hazard definition.
- 13.3 Every chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. Please refer to Appendix L in GLNPO's *Health, Safety and Environmental Compliance Manual* (May 1997, or as amended) for more detailed descriptions of the potential risks associated with any chemicals used in this method. It is good laboratory practice to wear a lab coat, safety goggles and gloves at all times.
- 13.4 It is the responsibility of the user of this method to comply with relevant chemical disposal and waste regulations as cited in GLNPO's *Health, Safety and Environmental Compliance Manual* (May 1997, or as amended). All applicable safety and waste handling rules are to be followed. Good technique includes minimizing contaminated waste.
- 13.5 Over-board discharges of chemical wastes are forbidden.

14.0 REFERENCES

- 14.1 "Methods for Chemical Analysis of Water and Wastes," March, 1979. EPA Publication #600/4-79-02.
- 14.2 "Operating Instructions," YSI Model 3200 Conductivity Meter.
- 14.3 "Calibration of Conductance Cells at 25°C with Aqueous Solutions of Potassium Chloride", April, 1959. Journal of the American Chemical Society. 1557-1559.
- 14.4 "Operating Instructions", HACH turbidimeter Model 2100N